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(54) **Liquid-liquid extraction column**

(57) A liquid-liquid extraction column through which a carrier liquid and an extraction agent are fed in counter-flow has alternate mixing zones (9) and passive zones (10) along its length. An axial rotary shaft (7) bears agitator paddles (11) in the mixing zones (9). The passive zones (10) are formed by baffles (12) which have generally axial ducts (17) to permit axial flow but offer a high resistance to radial flow. This prevents back-flow of liquid from a mixing zone (9). In operation, the extraction agent rises. In mixing zones (10) it forms a disperse phase in the carrier liquid. In the passive zones it tends to coalesce on the baffles (12) (which are of wettable material, e.g. PTFE), thus partially closing the ducts (17), which remain open to the extent required by the carrier throughflow. Thus the baffles adjust automatically to the flow rate. Droplets break away from the coalesced layers (18) and are redispersed in the above mixing zone (9).

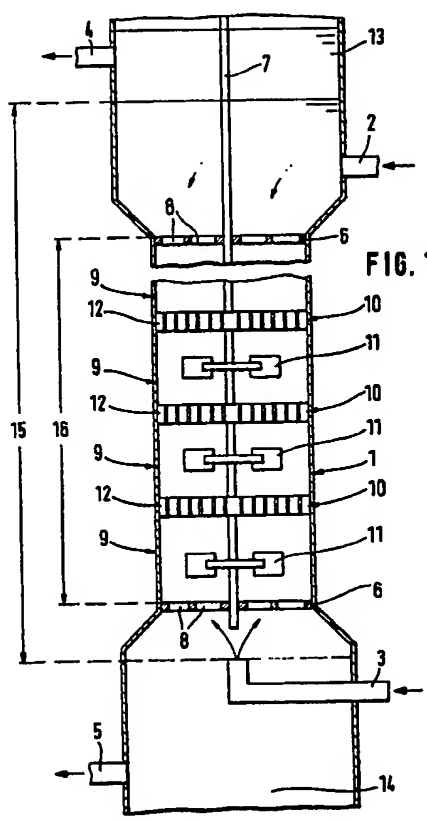

FIG. 1

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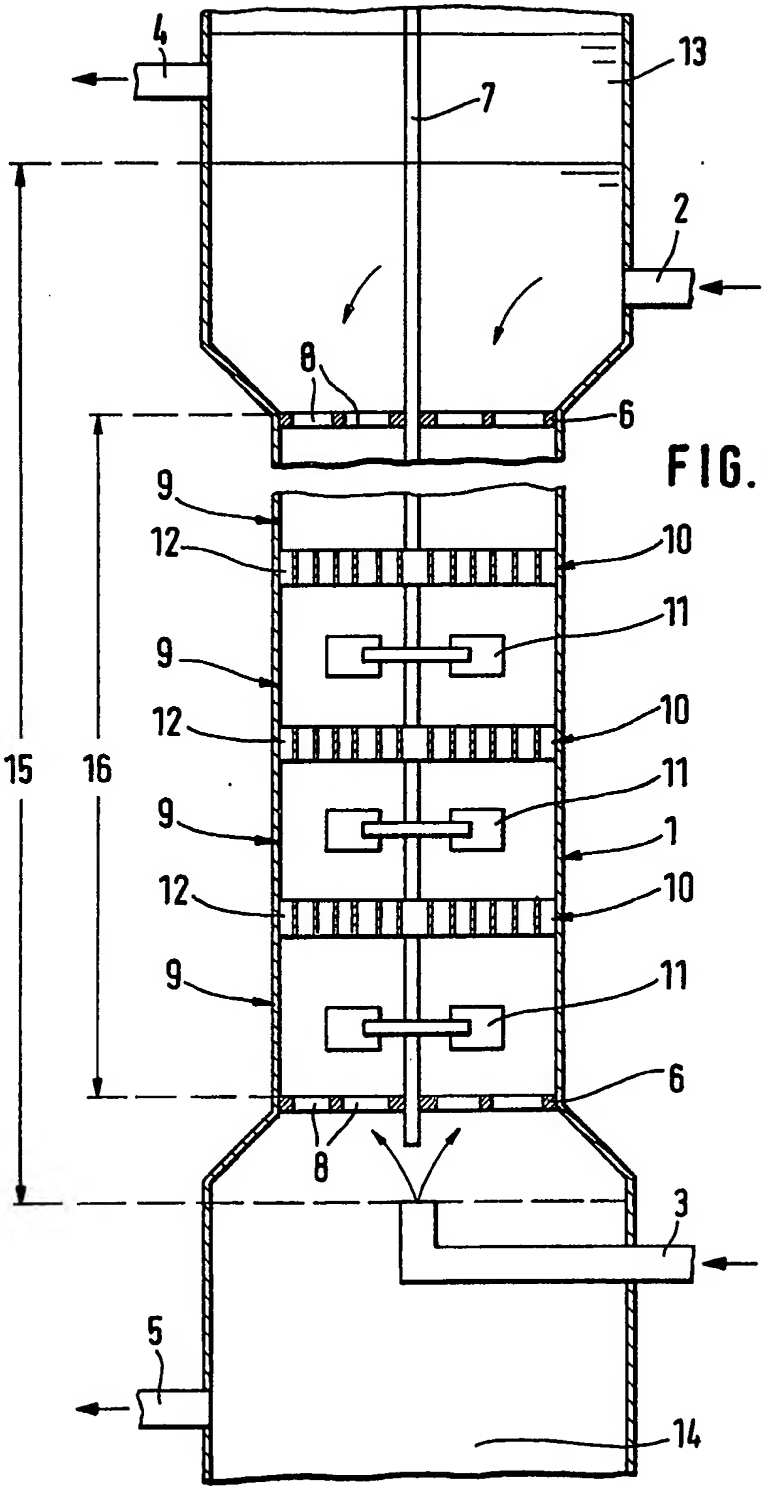

FIG. 1

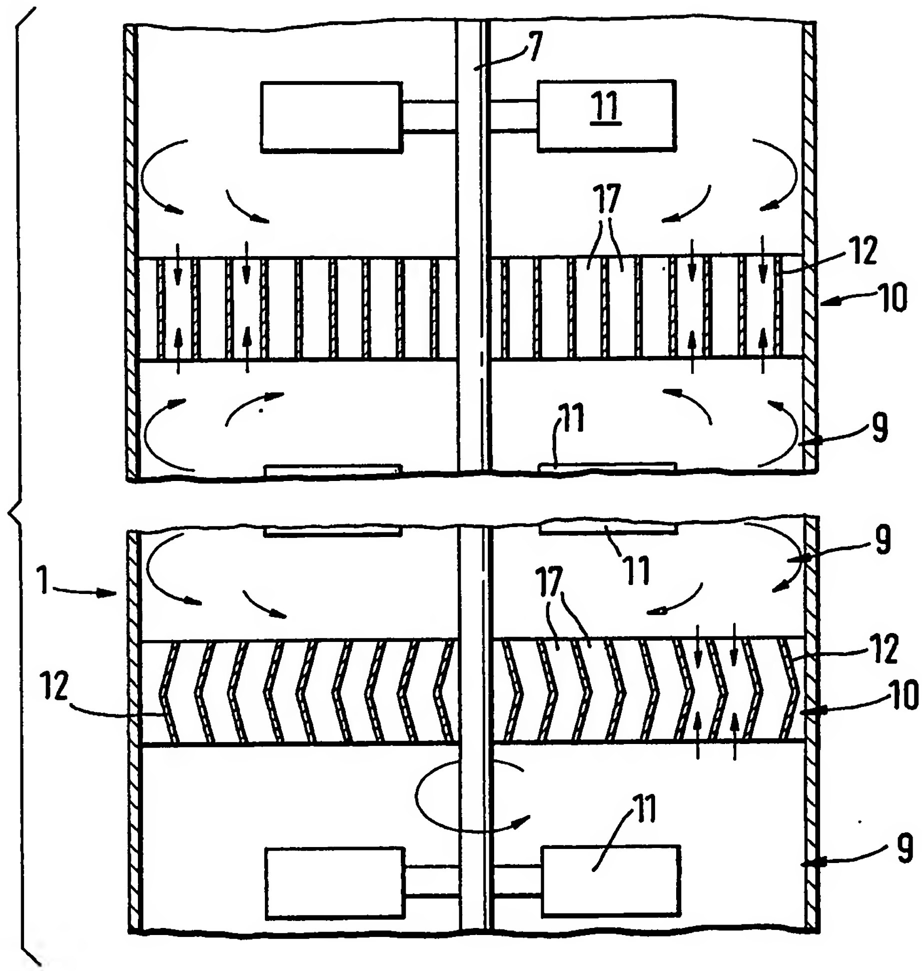

FIG. 2

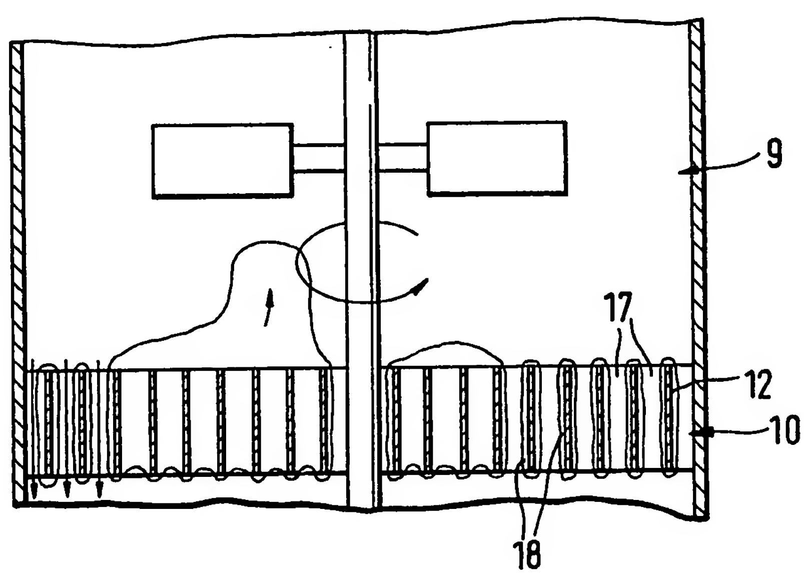

FIG. 4

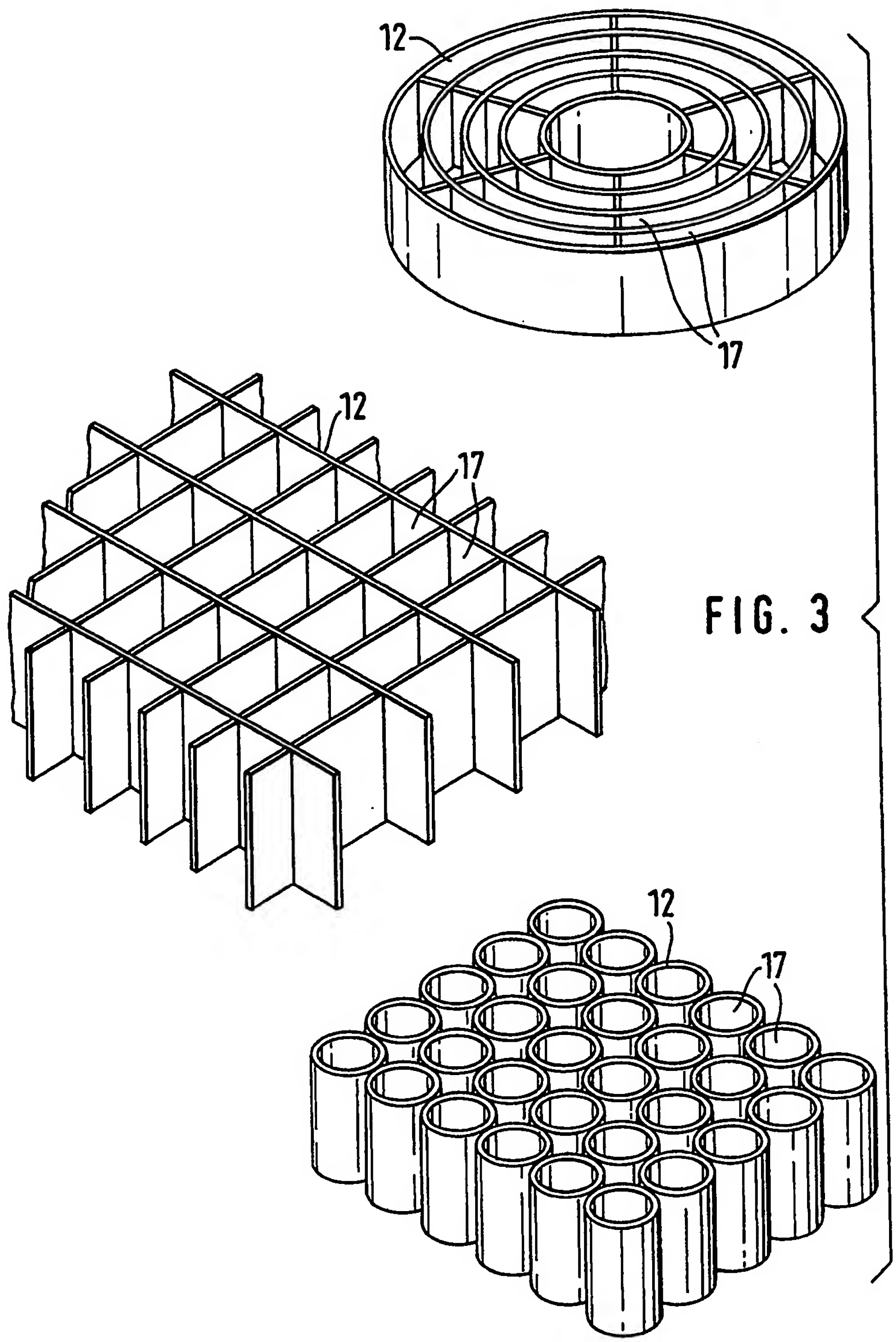

FIG. 3

SPECIFICATION

Liquid-liquid extraction column

The present invention relates to a liquid-liquid extraction column such as may be useful for, e.g. washing-out, two-phase chemical reactions, or neutralisation.

Extraction from a liquid phase is a separation process which is becoming increasingly widesread in chemistry, pharmacy and metallurgy. It has in the past tended to be used when other separation processes such as simple or fractional distillation, or rectification, have been found not very suitable for reasons such as great temperature sensitivity of the material or inability to attain a satisfactory approximation to the equilibrium curve, e.g. for fractionation.

In liquid-liquid extraction, the material to be separated off (the product) is generally initially contained in a "carrier liquid". The solvent which receives the material to be separated off is called the "extraction agent". The carrier liquid and the extraction agent must be of low or zero mutual solubility, and must differ in density. If the product is to be obtained out of solution, it is necessary to select an extraction agent from which it can subsequently readily be separated, for example by evaporation of the solvent.

In the extraction column, the carrier liquid or the extraction agent when they contain "product" material are known respectively as the "raffinate phase" and the "extract phase". In general, the raffinate phase should be eventually discharged with the lowest possible residual content of the product, whereas the extract phase should be product-rich. The rate with which the raffinate and extract phases approach equilibrium on contact depends largely on the diffusion velocity.

For extraction from a liquid phase, there are two classes of known counter-current extraction columns, which do or do not need to be supplied with mechanical energy. Those with such energy inputs are at present preferred since they can be smaller and of higher output. In such extraction columns, at least one of the phases (the disperse phase) is subjected to the action of agitators or pulsators so that new surfaces are continually being exposed to the continuous phase, thus accelerating the diffusion of the product material from the raffinate phase into the extract phase. This can increase the effective capacity of the column.

There are at the present day no generally applicable counter-current extraction columns. The known or proposed constructions are always designed for a closely specified capacity and field of use, and can be adapted only poorly or not at all to other conditions.

Desirably, a counter-current extraction column should have a high separation capacity for the widest possible range of capacities (throughputs and loading levels). A parameter related to these properties is the "separation volume", i.e. the volume of the extractor necessary for a separation capacity of a theoretical stage at a total throughflow (the sum of both phases) of 1 m^3/h.

The present invention is primarily concerned with extraction columns which must be supplied with mechanical energy. An example of a column of this general type is disclosed in "Techniklexikon", volume 35, page 132 (Rowohlt-Taschenbuchverlag GmbH); this is known as the RDC-Extractor. It uses a vertical column which has along its axis a stirrer shaft bearing horizontal stirring discs in the centre of a mixing zone. Arranged offset thereto at the column wall are stator rings the apertures of which are larger than the stirring disc diameter. The column has the disadvantage that back-mixing (return of a portion of a phase to a region of the column through which it has already passed, and dispersion of it therein) increases with increasing speed of the agitator discs, whereby the separation capacity is limited.

According to the invention there is provided liquid-liquid extraction apparatus having an elongate column for passage therethrough in counterflow of a carrier liquid which carries a substance to be extracted and an extraction agent, the column having alternate mixing and passive zones, the passive zones comprising flow restricting means for offering a substantially greater flow resistance to radial flow than to axial flow.

A suitable embodiment of the invention can be an extraction column which has a high flow limit and, even at high loading levels, has a high separation capacity.

Some embodiments of the invention will now be described with reference to the accompanying diagrammatic drawings, wherein:—

Figure 1 shows an extraction column in elevation;

Figure 2 is an enlarged detail of Fig. 1 showing two different types of insert in use:

Figure 3 shows perspective views of three examples of the inserts; and

Figure 4 shows a part of Fig. 1 drawn to a larger scale.

The extraction column 1 shown in Fig. 1 has respective upper and lower inlets 2 and 3 for the carrier liquid and the extraction agent, and respective upper and lower outlets 4 and 5 for the extract and the raffinate. Two shaft supports 6 are arranged in the extraction column 1 for supporting an agitator shaft 7 which is mounted to be rotatable by a motor (not shown). The shaft supports 6 are formed with apertures 8 to allow easy passage of the liquid phases. Between the shaft supports 6, the interior of the extraction column 1 is divided into alternate active mixing zones 9

and "passive" zones 10. In the active mixing zones 9, agitator paddles 11 having radial blades are secured to the shaft 7. The passive zones 10 contain baffles 12 secured to the column 1, the baffles being permeable to the liquid phases in the longitudinal direction of the extraction column 1.

In operation, the extraction agent (solvent) and the carrier liquid (which is denser, and bears in solution the substance to be extracted, i.e. the product) flow continuously into the column through their respective inlets 3 and 2. Because of the difference in density between the carrier liquid and the extraction agent, the latter flows upwardly, whereas the carrier liquid flows downwardly. At the same time the material to be extracted diffuses out of the carrier liquid into the extraction agent. The latter accumulates at the upper end of the extraction column 1 as an extract 13, whereas at the lower end the carrier liquid, freed (wholly or partially) from the material to be extracted, accumulates as a raffinate 14. In a zone 15 between the extract 13 and the raffinate 14, where mixing of the carrier liquid and the extraction agent occurs, both phases contain significant amounts of the material to be extracted. In this zone 15, the carrier liquid is called the "raffinate phase", and the extraction agent is called the "extract phase". Between the shaft supports 6 in a zone 16, the extract phase is dispersed in the raffinate phase by the agitator paddles 11 and forms the disperse phase; the non-dispersed raffinate phase flowing downwardly constitutes the continuous phase.

Fig. 2 shows in the upper half a first embodiment of an element insertable in the column to serve as a baffle 12. This has a plurality of channels or ducts 17 the length of each being several times the minimum cross-sectional dimension. The ducts 17 are parallel to the agitator shaft 7 and are thus generally parallel to the longitudinal axis of the extraction column 1; they will generally extend vertically in use. They provide in the direction of the longitudinal axis of the extraction column 1 a small flow resistance for the liquid phases, and in the radial and tangential direction a large flow resistance for the liquid phases. Consequently, there may take place in a mixing zone 9 intensive mixing, without a flowback of a significant amount of the disperse phase to an adjacent zone 9 occurring, since the mixing involves non-axial motion. The insert 12 shown in the lower part of Fig. 2 has ducts 17 which are angled relative to the longitudinal axis of the extraction column 1. They could alternatively be curved so as to appear concave relative to the column axis. The length of the said ducts 17 is again several times the smallest cross-sectional dimension. The curved or angled shape of the ducts 17 can give a still greater ratio of radial or tangential to axial flow resistance than in the case of the straight channels just described.

Referring to Fig. 3, the inserts for forming the baffles 12 may define ducts whose cross-sections are portions of annuli, square, or circular.

Alternatives which are not illustrated include hexagonal or elliptical cross-sections. A column may contain ducts of different forms. Preferably the duct length is substantially larger than the minimum cross-sectional dimension. It may be a multiple thereof. Furthermore, it is highly preferable for the sum of the duct cross-sections of a baffle to be at least 70% and preferably more than 90% of the internal cross-section of the extraction column 1.

The baffles 12 are preferably manufactured from or covered with a material which is wetted by the disperse phase. This has the advantage that the coalescent disperse phase in the region of the passive zones 10 tends to deposit on the walls of the ducts 17 (cf. Fig. 4) and to form there relatively large, coherent deposits 18. These form drops which are torn off from time to time and ascend into the mixing zone 9 located thereabove where they are once again dispersed. The wettability of the installations 12 improves to a considerable extent the coalescence in the passive zones 10, which in combination with the renewed dispersal accelerates the diffusion between phases of the substance in the following mixing zone. A preferred surface material for the baffles 12 comprises a self-lubricating plastics material (for example "Teflon" Registered Trade Mark).

The disperse phase ascending out of the lowermost mixing zone 9 (Fig. 1) passes into the first passive zone 10 where it tends to coalesce and precipitate on the walls of the baffle 12. The ducts 17 are gradually narrowed by the liquid deposits 18 (Fig. 4) and remain open only to the extent necessary for the throughflow of the downwardly flowing continuous phase (raffinate phase). In high capacity operation, i.e. with large feed rates of carrier liquid and extraction agent, relatively large through-passage cross-sections in the zone of the installations 12 remain open for the continuous phase; in operation at lower capacity, smaller passage cross-sections of the ducts 17 remain open. Thus, the baffles 12 have effective through-flow cross-sections which vary automatically with the mode (capacity) of use. Since it is generally true that for a maximum separation effect the disperse phase should, to the greatest possible extent, coalesce in the passive zones, and be dispersed again in the mixing zones, it is obvious that the automatic regulation of the through-flow cross-section of the baffle ducts 17 can lead to a good separating ability of the column for a wide range of operating capacities. This is further assisted by the hindering or prevention

of back-mixing resulting from the different axial and radial flow resistances of the baffles 12.

CLAIMS

1. Liquid-liquid extraction apparatus having an elongate column for passage therethrough in counterflow of a carrier liquid which carries a substance to be extracted and an extraction agent, the column having alternate mixing and passive zones, the passive zones comprising flow restricting means for offering a substantially greater flow resistance to radial flow than to axial flow.

2. Extraction apparatus according to Claim 1, wherein the flow restricting means are wettable by the extraction agent.

3. Extraction apparatus according to Claim 1, wherein the flow restricting means are wettable by water and/or organic solvents.

4. Extraction apparatus according to any one of Claims 1 to 3, wherein each flow restricting means is traversed by a respective plurality of generally axially extending ducts, each said duct being straight and parallel, or bent at an angle, or curved relative to the column axis.

5. Extraction apparatus according to Claim 4 wherein the total effective cross-sectional area of the ducts in any flow restricting means is at least 70% of the column cross-sectional area.

6. Extraction apparatus according to Claim 5, wherein said total effective cross-section is more than 90% of the column cross-sectional area.

7. Extraction apparatus according to any one of Claims 4 to 6, wherein at least a plurality of the ducts have polygonal or circular, or part-annular cross-sections.

8. Extraction apparatus according to any one of the preceding claims having a centrally axially extending rotatable mixer shaft from which agitator paddles project generally radially in the mixing zones.

9. Liquid-liquid extraction apparatus constructed and arranged substantially as described herein with reference to and as illustrated in the accompanying drawings.

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